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Carbon Dioxide Capture by Functionalized Graphene Oxide Adsorbent

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CARBON DIOXIDE CAPTURE BY FUNCTIONALIZED GRAPHENE OXIDE ADSORBENT

by
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ABSTRACT

The accumulation of CO_2 in the atmosphere is a major environmental hazard. As a result various methods for capturing CO_2 was suggested. One of such methods included the development of carbon-based adsorbents due to their large surface area and low heat capacity for CO_2 capture which was studied in this report. Graphene oxide (GO) was selected as the adsorbent for its potential benefit in CO_2 capture.

A modified hummers method was used for the synthesis of GO which showed the existence of the hydroxyl (3207 cm^{-1}), epoxyl (1036 cm^{-1}) and carboxyl (1720 cm^{-1}) functional groups in its FTIR spectra. The C/O ratio in the GO was on an average of 2.57. The hydroxyl groups were subjected to a Johnson-Claisen rearrangement which gave rise to an ester functional group attached to GO. The ester group was saponified to carboxylic acid groups and reacted with ethylenediamine to produce an amine-functionalized GO. The amine which increases the CO_2/H_2 selectivity for pre-combustion CO_2 capture got attached to the carboxylic and epoxyl groups in GO. Thermogravimetric analysis was done on the amine-functionalized GO and a weight gain of about 1.173% which signified the adsorption of CO_2 was achieved. However, improvements to this procedures need to be taken since a considerable amount of CO_2 was not captured in future work.

ACKNOWLEDGEMENTS

I would like to thank my family for their continuous love and support throughout my life and academic career. I offer my sincere gratitude to my advisor, Dr. Chen for giving me the opportunity to participate in his research, Dr. Stefanie Sydlik from Carnegie Mellon University and Dr. Mattern for their continued assistance. Thank you to Dr. Egiebor and Dr. Williford for taking the time to read this thesis. Lastly, I'd like to thank all my friends and professors for their support and encouragement during my four years at Olemiss.

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CHAPTER 1

INTRODUCTION

The 20th century has seen the rapid increase of population and explosive growth in energy consumption. Currently, over 85% of world energy demand is supplied by fossil fuels [18]. Environmental issues due to emissions of pollutants from combustion of fossil fuels have become global problems, including greenhouse gases (GHG) [18]. Increased levels of GHG in the atmosphere is believed to cause global warming [18]. Among these GHG, CO_2 is the largest contributor due to the amount present in the atmosphere contributing to 60% of global warming [17]. Various options to reduce total CO_2 emission into the atmosphere including development of technologies to capture CO_2 have been suggested [18].

A critical review conducted by Chen et al. suggested that carbonaceous compounds react with (CO_2 +H_2) mixture through photochemical, and photo-catalytic routes [3]. In the review, the interactions between CO_2 and carbonaceous materials, especially CO_2 fixation on carbon, which is vital for many CO_2 capture and utilization processes were discussed [3]. It was postulated that reductive photo-carboxylation of edge carbons of polycyclic aromatic hydrocarbon (PAH) could be an effective route for CO_2 capture as in the chemical equation below:

$$PAH \xrightarrow{h\nu} PAH^{*} + DMA \rightarrow PAH^{\bullet -} + CO_2$$
$$\rightarrow PAH^{\bullet} - CO_2^{-} + i\,\mathrm{Pr}OH \rightarrow H - PAH - CO_2^{-}$$

where DMA denotes N,N-dimethylaniline (an electron donor) and iPrOH, 2-propanol (a hydrogen donor), respectively [3].

Biochar (a byproduct of bio-oil production) was chosen in this study conducted by Chen et al. for various reasons including the reactivity of its edge carbons since it contains graphene and graphene oxide layers which could serve as binding sites for CO_2. Biochar was treated with ultrasound and light, in CO_2/H_2O at 60^0C and 1 atm. It was noticed that its carbon content increased by 13% which suggested a new route for CO_2 capture [3].

In order to improve this CO_2 capture process, graphene oxide which could be exfoliated from biochar during ultrasound and contains the reactive edge carbons with a large surface area for CO_2 capture and functionalization was suggested. Chen et al. proposed that functionalizing graphene oxide with amines which increases the CO_2/H_2 selectivity could induce higher pre-combustion CO_2 capture capacity. However, this procedure was not investigated and lead to the rationale for this thesis.

1.1 Graphene Oxide

1.1.1 Background of Graphene Oxide

Graphene oxide (GO) as a history that extends back many decades to some of the earliest studies involving the chemistry of graphite [5]. It was first discovered by British chemist B.C. Brodie in 1859 when exploring the structure of graphite by investigating the reactivity of graphite flakes [9]. Brodie's reaction involved adding potassium chlorate to a slurry of graphite in fuming nitric acid (HNO_3) which resulted in an increase in the overall mass of the graphite flake [5]. He found the material to be composed of carbon, hydrogen, and oxygen after combustion with a C:H:O composition of 80.13 :0.58 :19.29 and dispersible in pure or basic water but not in acidic media [5]. Various methods for the preparation of GO were proposed after Brodie's method which include the Staudenmaier process, the Hummers-Offeman process, and anodic oxidation of graphite electrodes in nitric acid [9].

Nearly 40 years after Brodie's discovery of the ability to oxidize graphite,

L.Staudenmaier improved the method with the addition of concentrated sulfuric acid to increase the acidity of the mixture and addition of potassium chlorate in multiple aliquots over the course of the reaction rather than in a single addition as Brodie had done [5]. This resulted in an overall extent of reaction similar to Brodie's approach with a C:O ratio of 2 :1 [5]. In 1957, Hummers and Offeman developed a safer, quicker, and more efficient process using a mixture of sulfuric acid (H_2SO_4), sodium nitrate ($NaNO_3$), and potassium permanganate ($KMnO_4$) which achieved similar levels of oxidation [15]. This process is widely used in producing GO often with some modifications in the world today [8].

1.1.2 Structure of Graphene Oxide

The exact chemical structure of GO has been the subject of considerable debate over the years and even to this day due to the complexity of the material which includes sample-to-sample variability [5]. This results from the nonstoichiometric atomic composition and lack of precise analytical techniques for characterizing GO [5]. Despite these obstacles, considerable effort has been put into understanding the structur of GO much of it great success [5].

Since GO was discovered in 1859, by Brodie, various structural models have been proposed including Hofmann's model, Ruess's model, Scholz-Boehm's model, and Lerf-Klinowski's model [9]. Hofmann first proposed that only epoxy groups were spread across the basal planes of graphite [5].

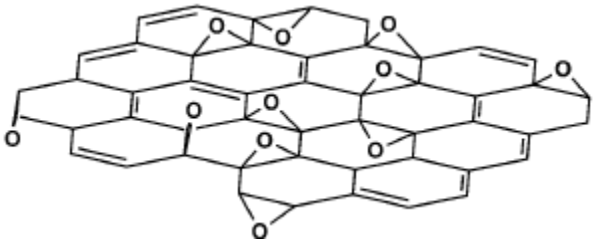

Figure 1.1. Hofmann's GO model showing only epoxy groups on the surface
From Dreyer et. al [5]

Although Hofmann's model showed the existence of epoxy groups, however it failed to provide information about other functional groups including hydrogen [9]. Ruess suggested a variation of this model which incorporated hydroxyl groups into the basal plane, accounting for the hydrogen content of GO [5]. He proposed that the carbon layers were wrinkled and consisted of trans-linked cyclohexane chairs [9].

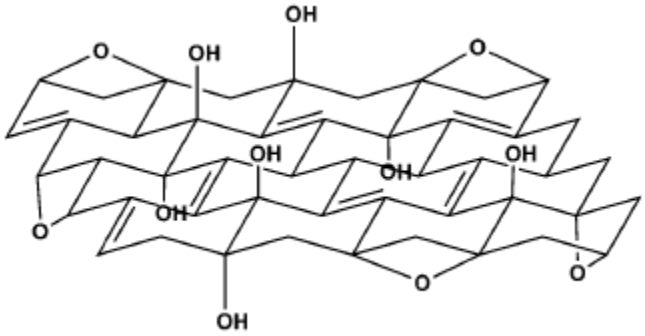

Figure 1.2. Ruess's GO model showing hydroxyl groups also on the surface
From Dreyer et. al [5]

Scholz and Boehm revised Ruess's model which proposed that ketone groups are present in GO and carbon layers were corrugated [9].

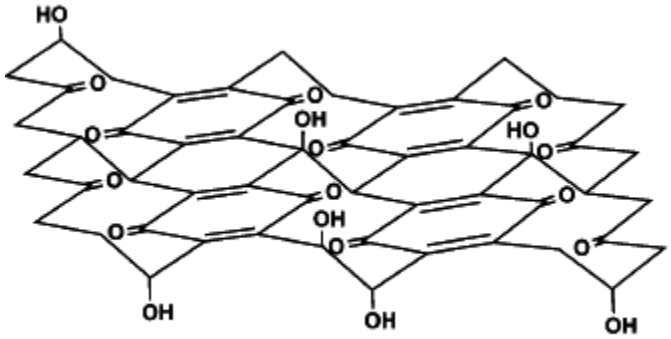

Figure 1.3. Scholz-Boehm's GO model showing ketone groups
From Dreyer et. al [5]

The most recent and well received model of GO was proposed by Lerf and Klinowski in 1998 [5]. Their model was based on solid-state nuclear magnetic resonance

(NMR) spectroscopy which suggested hydroxyl (-OH), and epoxy (-O-) groups exist in GO and carboxyl (-COOH) groups are located at the edges of layers [9].

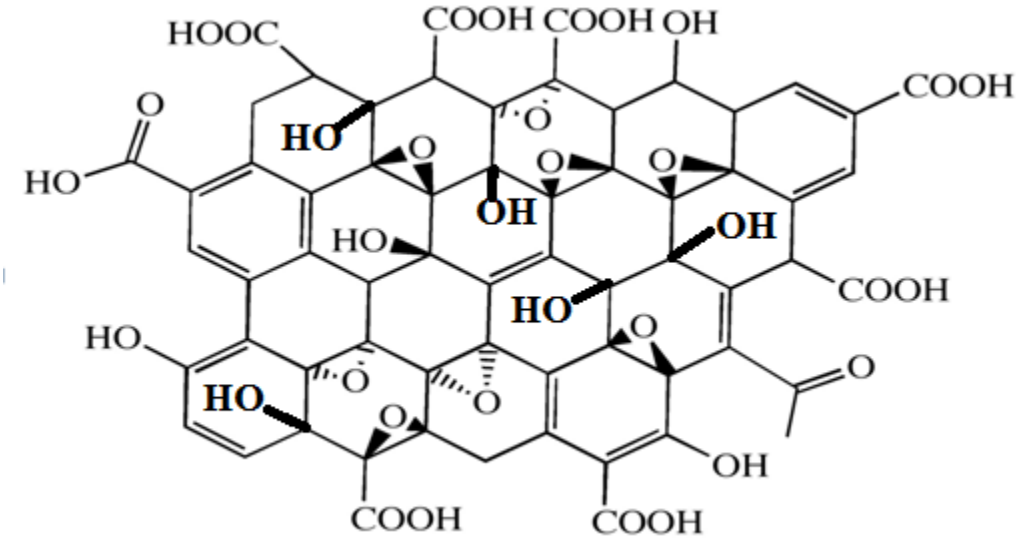

Figure 1.4. Lerf-Klinowski's GO model showing hydroxyl. epoxyl and carboxyl groups From Atanasov et. al [1]

GO is usually derived from the exfoliation of graphitic oxide, and hence is likely to share a similar structure [16]. The structure of GO is often simplistically assumed to maintain the hexagonal symmetry of a graphene sheet bonded to oxygen in the form of carboxyl, hydroxyl and epoxy groups with a C:O ratio between 2.1 and 2.9 [15]. Hydroxyls and (1,2) epoxies are the dominant functional groups with carbonyls existing around the edges of the holes [6]. It has an average in-plane carbon-carbon spacing 0.1421 ± 0.0007 nm, indistinguishable from that of graphene with no regular ordering of the functional groups [16]. This is caused by variations in the degree of oxidation as a result of differences in graphite source [5].

1.1.3 Properties of Graphene Oxide

GO is easily dispersible in water and other organic solvents due to the presence of the oxygen functionalities [7]. This makes it easier to handle, functionalize, transport, and deposit [16]. GO is an insulator due to the disruption of its sp^2 bonding

networks, and almost a semiconductor with conductivity strongly dependent on the degree of oxidation or reduction [16].

1.1.4 Applications of Graphene Oxide

Graphene oxide, an oxidatively derived and sonochemically exfoliated form of graphite is known to be an excellent platform for the development of covalent surface and functionalization methods [4]. There are various applications of GO including graphene manufacture, coatings, paper manufacture, rechargeable battery electrode, and CO_2 capture depending on the functionalization method [15].

1) Graphene Manufacture: The hydrophilic nature of GO enables it to easily disperse in water and break up into macroscopic flakes mostly one layer thick which is chemically reduced to give a suspension of graphite flakes [15]. These flakes are subjected to partial reduction with hydrazine hydrate at 100^0C for 24 hours by exposure to a strong pulse of light which produces graphene [15]. Rapid heating (greater than 2000^0C) of GO to 1050^0C also produces graphene sheet due to the release of CO_2 as the oxygen functionalities are removed [15].

2) Coatings: One of the major uses of graphene oxide is in the production of conductive films. GO films can be deposited on essentially any substrate, and later converted to a conductor [13]. Optical films made from GO are impermeable under dry conditions. Graphene made from GO could be used to coat glasswares which can be used as containers for corrosive acids [15].

3) Paper Manufacture: GO mixes readily with many polymers, forming nanocomposites, and greatly enhances the properties of original polymer; which includes elastic modulus, tensile strength, electrical conductivity, and thermal stability [13]. GO flakes that have been dispersed could be sifted and pressed to make a strong GO paper [15].

4) Rechargeable Battery Electrode: GO could be used as a free-standing bat-

tery anode material for room temperature lithium-ion and sodium-ion due to its high surface area [15].

5) CO_2 Capture: GO is susceptible to amine functionalization and then CO_2 capture. Amine functionalized GO show high CO_2 adsorption capacity [2]

1.2 Objective and Scope of Current Work

The accumulation of CO_2 in the atmosphere causes climate change which is a severe environmental problem faced in the world [2]. Adsorption on solid adsorbents, in particular carbon-based adsorbents is considered to be one of the most efficient ways to capture CO_2. This is due to their chemical inertness, low cost and high surface area [2].

The objective of this work is to develop new carbon-based adsorbents to improve CO_2 capture at low cost in an energy-efficient manner. Due to the slightly acidic nature of CO_2, the basicity of the adsorbents plays an important role in achieving high CO_2 capture performance [2]. GO would be used as the building block for developing adsorbents due to its large surface area and chemical stability for capturing CO_2 and functionalization. Amine functionalization of GO would be a desirable step because it increases the CO_2/H_2 selectivity for pre-combustion CO_2 capture.

In this work, GO was prepared using a modified hummers method as seen in the experimental section. The hydroxyl functional group in GO was converted to ester groups via a Johnson-Claisen rearrangement. In this rearrangement, GO reacted with triethyl orthoacetate to give carbon bonded reactive ester functionalities on the graphitic framework. This ester-functionalized GO was reduced to carboxyl groups using sodium hydroxide.

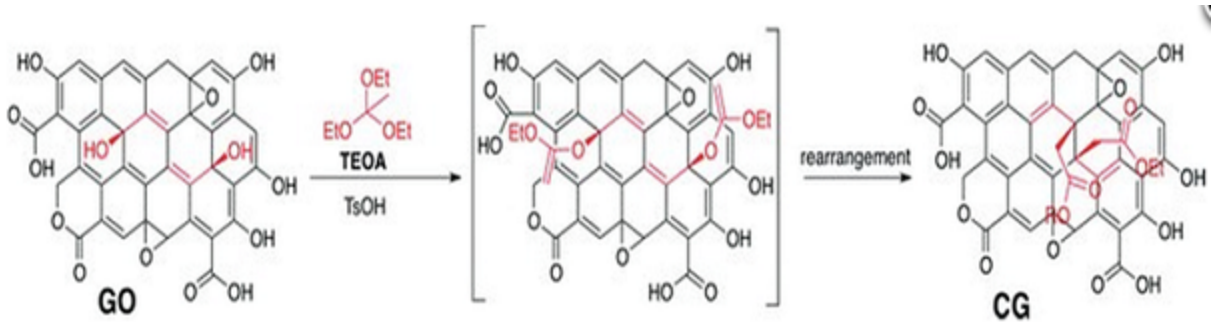

Figure 1.5. Johnson-Claisen Rearrangement of GO
From Sydlik et. al [12]

After saponification, GO reacted with an amine (ethylenediamine) to aid the capture of CO_2. The amines get attached to the carboxyl and epoxyl groups on GO.

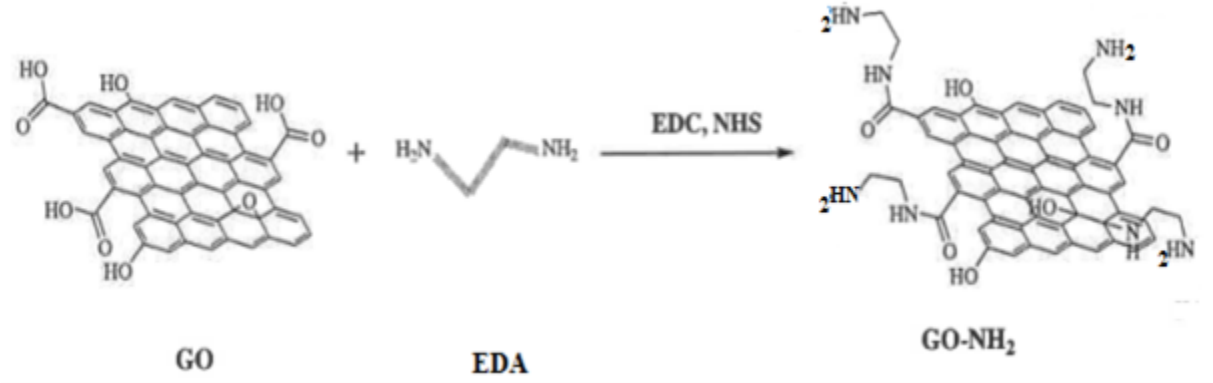

Figure 1.6. Amine-Functionalized GO
From Shang et. al [11]

CHAPTER 2

EXPERIMENTAL SECTION

2.1 Materials

Graphite powder (micro450) was used as received from Asbury. H_2SO_4 (98 mass%), $NaNO_3$, $KMnO_4$, H_2O_2 (30 mass%), distilled water, ethanol, triethyl orthoacetate (98 mass%), para-toluene sulfonic acid, NaOH (1M), acetone, methanol, n-hydroxysuccinimide, 1-ethyl-3 carbodiimide, ethylenediamine, were used as received. HCl (30%) was prepared in the lab. A salt bath was used during reflux due to the high temperature required.

2.2 Synthesis of Graphene Oxide

As stated in [10]:

Synthesis was accomplished by an modified Hummers method. Concentrated H_2SO_4 (23 mL) was added to 1 g of graphite powder and 0.5 g of $NaNO_3$. The mixture was cooled using an ice bath to 0^0C. Potassium permanganate (3 g) was added slowly in portions to keep the reaction temperature below 20^0C. The reaction was warmed to 35^0C and stirred for 7 hours. Additional $KMnO_4$ (3 g) was added in one portion, and the reaction was stirred for 12 hours at 35^0C. The reaction mixture was cooled to room temperature and 140 mL of distilled water with 1 mL hydrogen peroxide (H_2O_2) was added. The mixture was centrifuged (5000 rpm for 10 mins) and the supernatant decanted away. The remaining solid material was then centrifuged in succession with distilled water, 30% hydrochloric acid (HCl) and twice with ethanol. The product was vacuum-dried overnight at room temperature, obtaining 1.50 g of solid product.

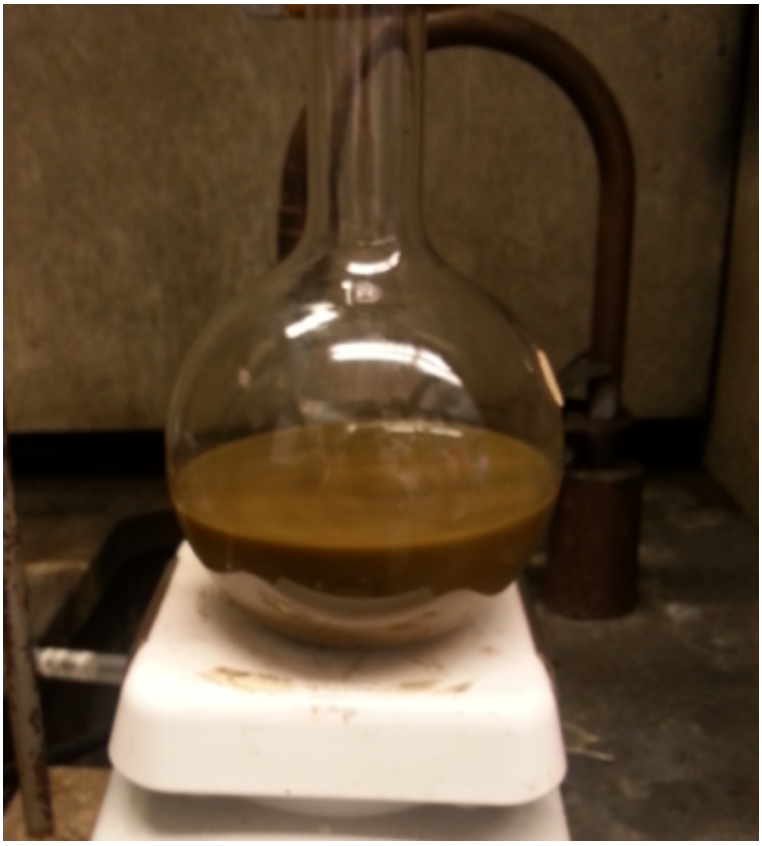

Figure 2.1. GO after the addition of H_2O_2

2.3 Synthesis of Claisen Graphene Oxide

As stated in [12]:

Synthesis was accomplished by the Johnson-Claisen rearrangement. Graphene Oxide (0.37 g) was added to 75 mL of triethyl orthoacetate. The GO was dispersed via 10 min of sonification.

Figure 2.2. Claisen GO during sonification

Catalytic para-toluene sulfonic acid (0.01 g) was added in one shot to the mixture. The reaction was allowed to proceed at reflux (150^0C) for 36 hours which converted some of the hydroxyl groups to ester groups. This would be called CG1. To favor the formation of carboxylic acid groups, 75 mL of 1M sodium hydroxide (NaOH) in ethanol (bp 78^0C) was added to CG1. The reaction was allowed to cool to room temperature while stirring vigorously for 3 hours. The reaction mixture was centrifuged (5000 rpm for 10 min) and the supernatant discarded. This was repeated three times with water and twice with acetone. The solid residue was vacuum-dried overnight at room temperature to yield 0.3 g of product which would be referred to as CG2..

Figure 2.3. Claisen GO during reflux with a salt bath

2.4 Amine Functionalization

As stated in [14]:

CG2 (0.1 g) was added to 150 mL of water and stirred. N-hydroxysuccinimide (0.25 g) and 0.25 g of 1-ethyl-3 carbodiimide (EDC) was added to the reaction mixture and stirred for 24 hours at room temperature. The mixture was centrifuged and the supernatant discarded. The solid residue was suspended in 150 mL methanol and 1 g of ethylenediamine (EDA) was added. The reaction mixture was allowed to stir for 72 hours. The mixture was centrifuged three times each with methanol and acetone. The solid product was vacuum-dried at room temperature to yield 0.08 g of product.

Figure 2.4. Addition of EDA to CG2

2.5 Characterization

Fourier transform infrared spectroscopy (FTIR) spectra were determined using an Alpha FT-IR spectrometer using the OPUS software package. Thermogravimetric analysis (TGA) was performed using a TA instruments Q500-1475 under helium at a scan rate of 20^0C/min to 60^0C.

CHAPTER 3

RESULTS AND DISCUSSION

3.1 Synthesis of Graphene Oxide

Graphite was oxidized using two methods; hummers and modified hummers method and observed to see which one gave better synthesis.

3.1.1 Hummers method

The standard hummers method proposed by Hummer and Offeman in 1958 was used in this synthesis. This procedure is similar to the modified hummers method explained in chapter 2 with the exception of additional $KMnO_4$ and washing the product only four times. In hummers method, the product was washed multiple times until a stable pH was recorded as seen in Table 3.1

Number of Washes	pH
1	0.95
2	1.03
3	1.75
4	2.53
5	2.92
6	3.25
7	3.29
8	3.35
9	3.41
10	3.49
11	3.57
12	3.58
13	3.58
14	3.58

Table 3.1. pH of GO using Hummers Method

From the table, pH doesn't change after a certain amount of times washed, so any continous wash would make no difference in pH. The pH got stable at 3.58 which signified the acidic nature of GO. After purification, GO was characterized with FTIR as seen in Figure 3.1 but the peaks showed were not encouraging.

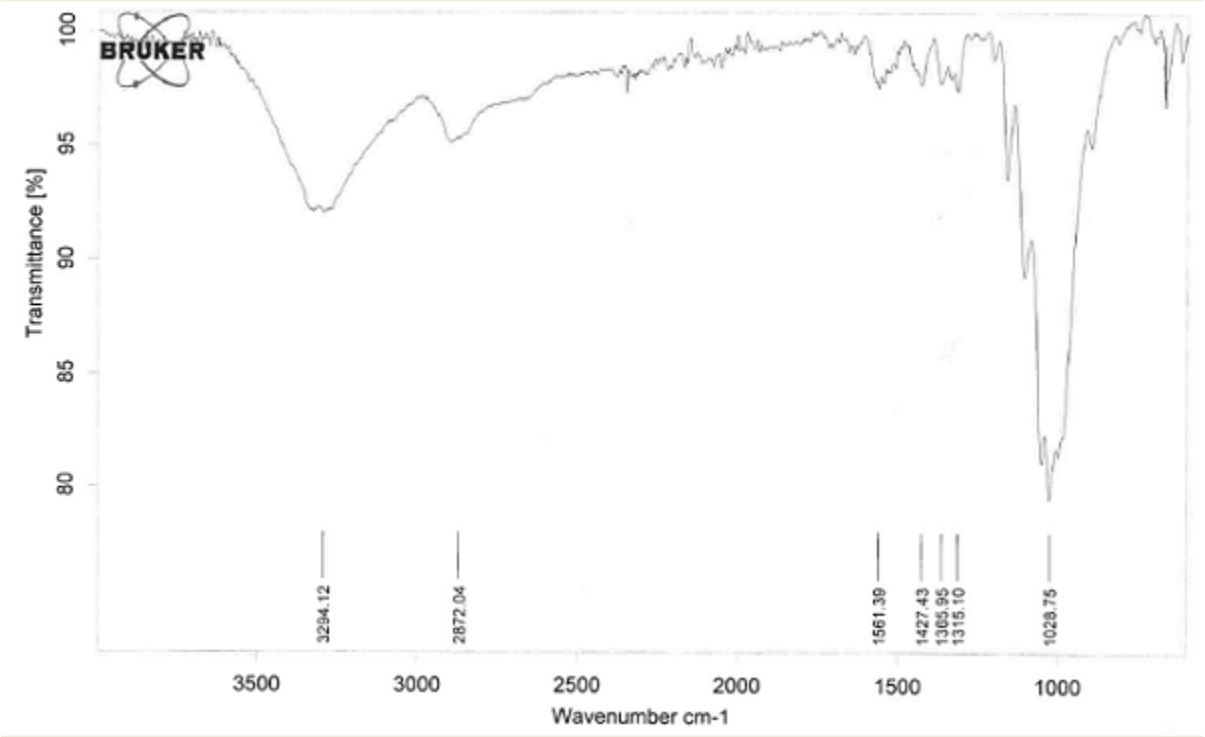

Figure 3.1. Characterization of Hummers GO using FTIR

A lot of disruptions were seen in this spectra which could have been due to the multiple times GO was washed which may have allowed moisture in the sample. Also, several small peaks were observed, however this result doesn't give convincing peaks for the functional groups expected in GO. For this reason, a modified hummers method was introduced to improve the procedure.

3.1.2 Modified Hummers Method

All synthesis were done in the fume hood. After the addition of $KMnO_4$, the mixture turned to a very thick dark color, however with the addition of H_2O_2 which was used to terminate the reaction, the mixture turned brown. The pH for this sample wasn't measured because it was observed from the GO produced using hummers method that the pH didn't affect the outcome of the product.

3.1.2.1 Changes in Sample Weight and C/O ratio of GO

The modified hummers method was used to produce a brown color (moderately oxidized) GO with varying weight and C/O ratio during vacuum-drying at 20^0C for 4 days as seen in Table 3.2.

Day	Weight Change	C/O ratio
1	1.5569	2.3942
2	1.5111	2.6088
3	1.5035	2.6481
4	1.5028	2.6518

Table 3.2. Changes in Weight and C/O ratio of GO during Vacuum-Drying

As seen in Table 3.2, the weight of GO decreases as it gets dried as a result of the loss of moisture after purification during drying. The weight of GO increased by about 0.5g as compared to the 1g of graphite started with due to the oxidation of GO. It should be mentioned that as the weight changes, the C/O ratio increases and stays between the known ratio (2.1-2.9) of GO.

3.1.2.2 Characterization

. The FTIR spectra for GO was recorded after purification. GO is expected to contain the hydroxyl, epoxyl, and carboxyl functional groups so a peak suggesting each of this groups should be seen in Figure 3.2.

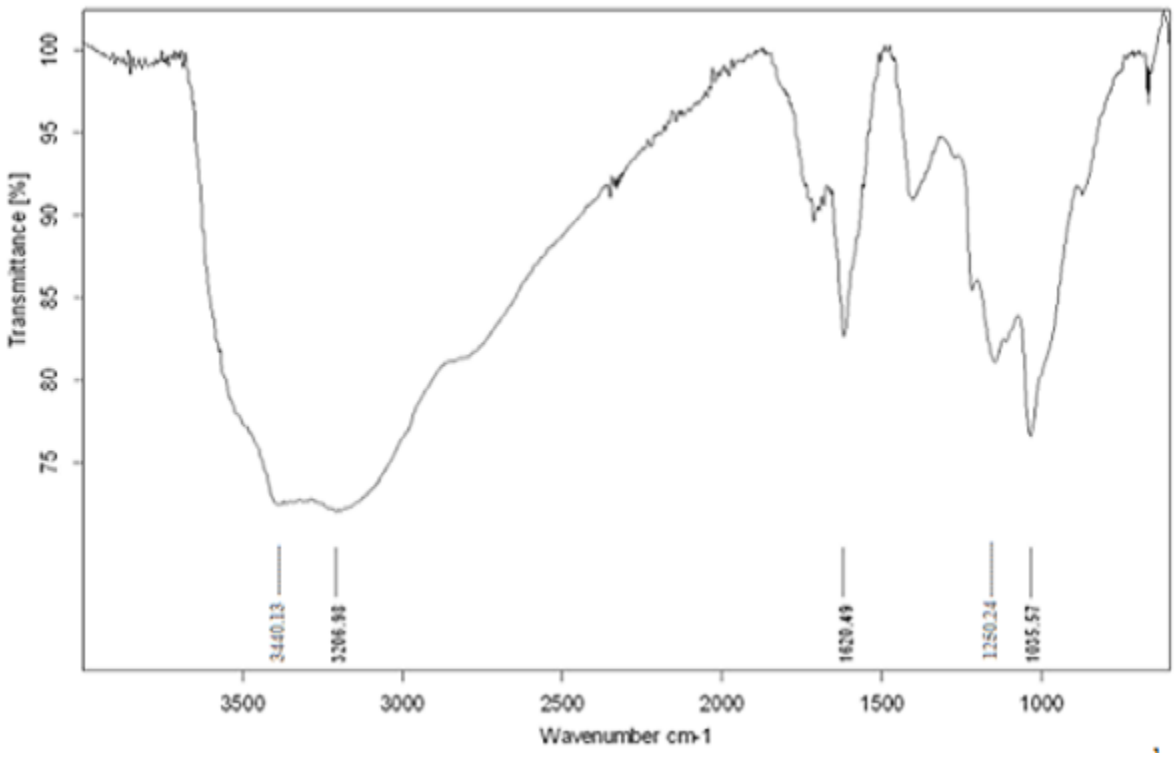

Figure 3.2. Characterization of Modified Hummers GO using FTIR

There are three sharp peaks centered at 3440, 1620, and 1036 cm^{-1}. The peak at 3440 cm^{-1} denotes the C-OH stretch. The peak at 1620 cm^{-1} corresponds to the C=C from unoxidized sp^2 CC bonds. The peak at 1036 cm^{-1} arises from epoxy groups. There are also small peaks at 1720 cm^{-1} which could denote the C=O stretch and 1250 cm^{-1} from C-O vibrations.

3.2 Synthesis of Claisen Graphene Oxide

GO made with hummers method was first used for this procedure, however it produced a fluffy Claisen GO which was indispersible in the solvent (water) used in the amine functionalization. As a result, moderately oxidized GO made with modified hummers method was used for this procedure since it was amphiphilic, which allows an easier dispersion in the organic conditions used. Fully oxidized GO (C/O ratio less than 2.1) required significant sonication to form a good dispersion in triethyl

orthoacetate (TEOA) due to its hydrophilic property, however moderately oxidized GO formed a good dispersion with just stirring. TEOA was used as the solvent for this reaction due to its high boiling temperature (142^0C) which was necessary for the extent of reaction. Para-toluene sulfonic acid was used as a catalyst to improve the extent of reaction, however due to the acidic nature of GO, a very small amount was used.

The Johnson-Claisen rearrangement helped convert some of the hydroxyl groups in GO to ester groups instead of carboxyl groups, so 1M NaOH in ethanol was added to aid this formation. Ethanol was used instead of water because water would phase separately from the TEOA, and would disrupt the reaction which made ethanol a better choice.

3.2.1 Characterization

The FTIR spectra gotten from CG1 showed an intense ester C=O peak at 1730 cm^{-1} as illustrated in Figure 3.3.

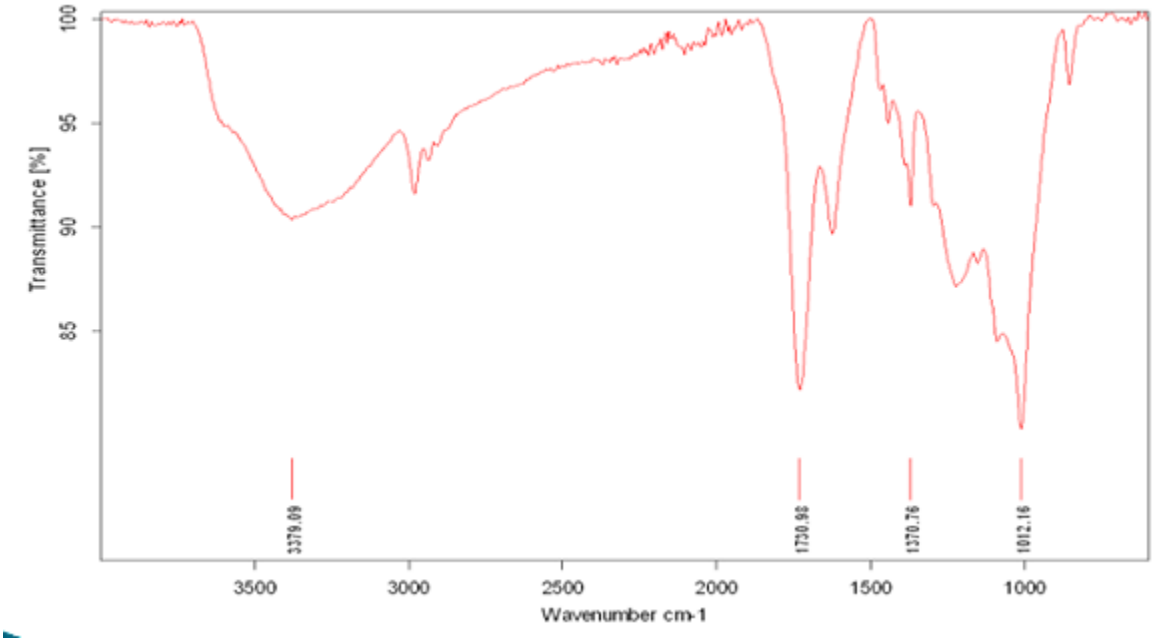

Figure 3.3. FTIR Spectra of CG1 showing ester C=O peak

Initially, GO was refluxed at 130^0C (below the boiling temperature of TEOA which is 142^0C) to produce CG2 but it was found out that the reaction would actually be unable to be refluxed at this temperature due its low reaction efficiency. This produced an unstable FTIR with a lot of disruptions as seen in Figure 3.4.

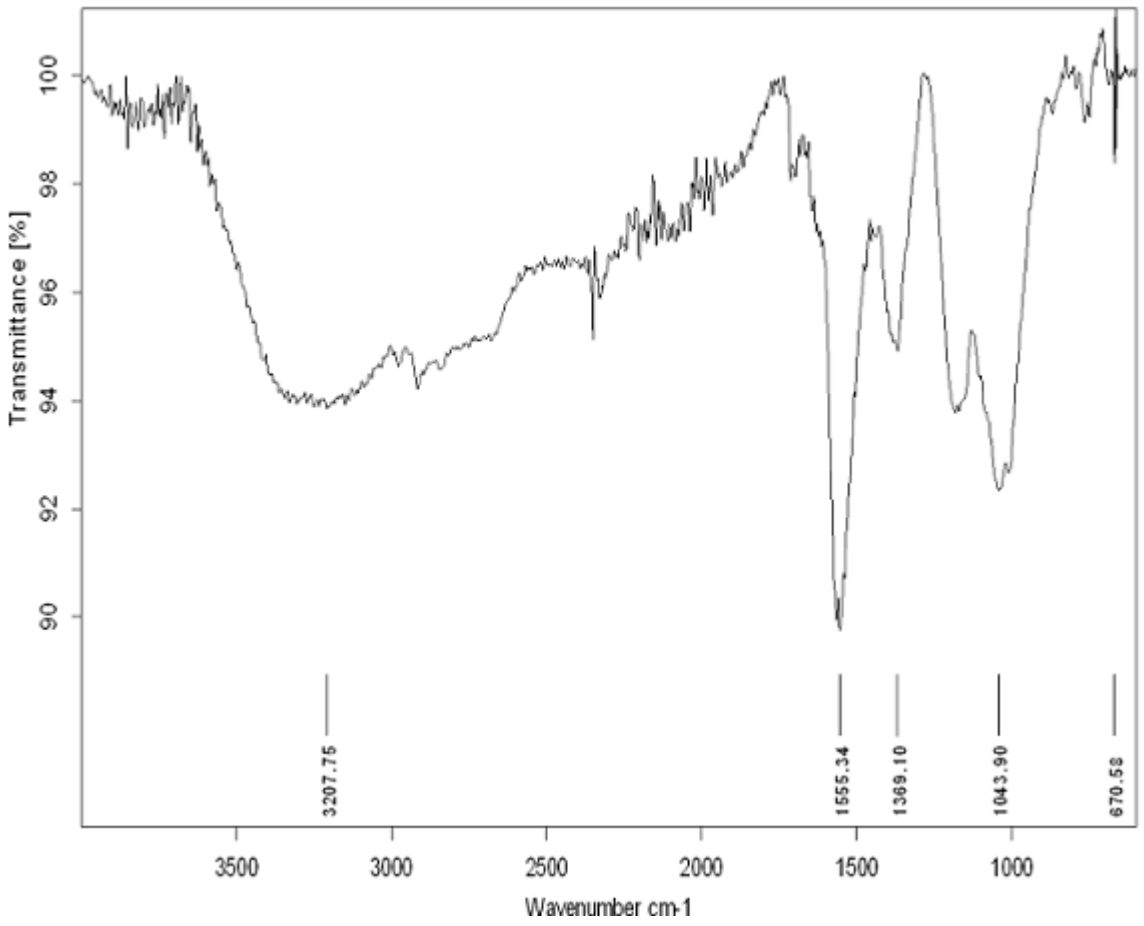

Figure 3.4. Characterization of CG2 at 130^0C reflux temperature

For higher reaction efficiency, reflux at a temperature greater than the boiling temperature of TEOA which was 150^0C was used to produce CG2 and characterized as seen in Figure 3.5.

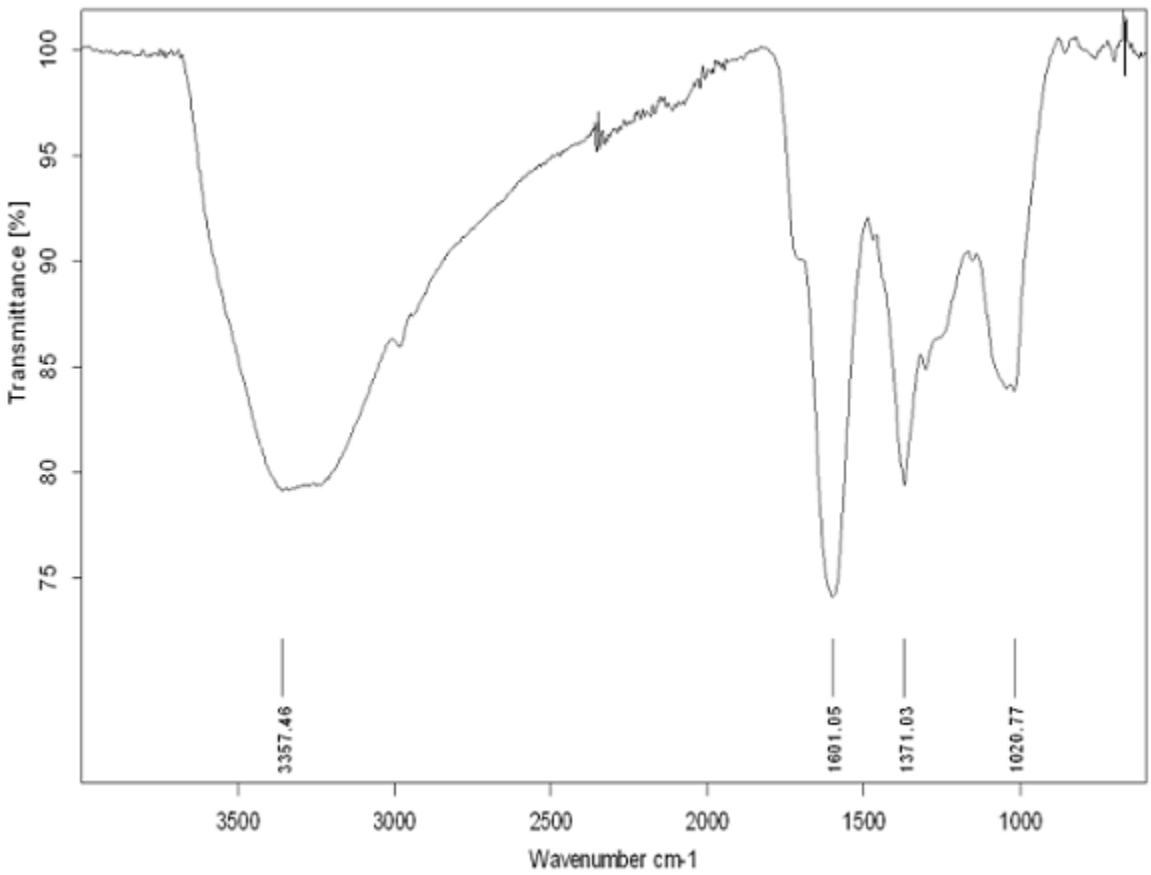

Figure 3.5. Characterization of CG2 at 150^0C reflux temperature

CG2 at 150^0C reflux showed an intense peak of the carboxylate C=O group at 1600 cm^{-1}.There are few disruptions seen between 2000 cm^{-1} and 2400 cm^{-1} which could be due to impurities in the form of some other functional groups not needed in Claisen GO.

3.3 Amine Functionalization

CG2 was further functionalized with amines to aid CO_2 capture. There are two methods that could be used to attach amines to CG2; using acid chlorides or EDC to convert acid groups in CG2 to amides. The latter option was chosen for this procedure due to it being more economical and safe since it doesn't involve toxic chemicals. The initial procedure for this option was to leave CG2 stirring with EDA for 24 hours which was called ACG2-1, however the FTIR showed in Figure

3.6 didn't show substantial results that amine could have been attached. The peaks looked similar to the CG2 spectra peaks and there was no definite way to distinguish if the peak shown at 1564 cm^{-1} belonged to the amine group (1560-1640 cm^{-1}) or some other group like the carboxylate (1550-1610 cm^{-1}).

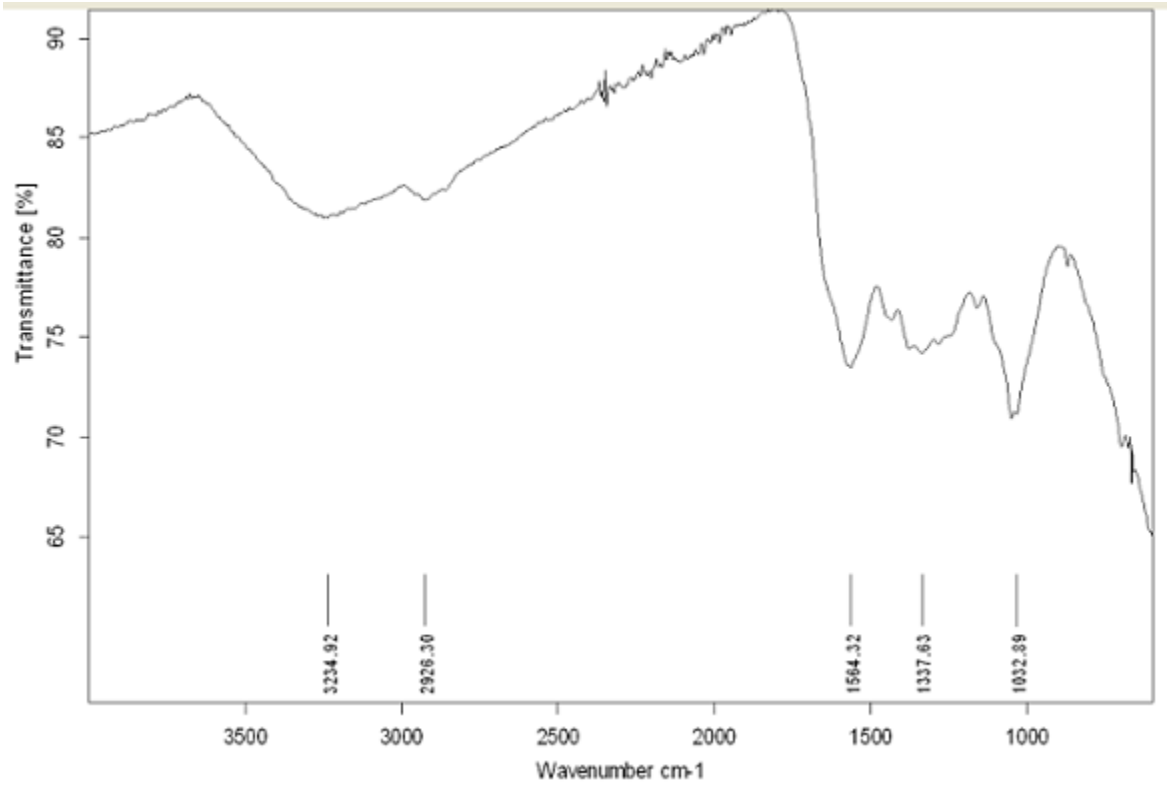

Figure 3.6. FTIR of Amine-Functionalized CG2 (ACG2-1) stirred in EDA for 24 hrs

As a result, the procedure was modified by allowing it to stir for 72 hours which was referred to as ACG2-2 and characterized with FTIR as illustrated in Figure 3.7.

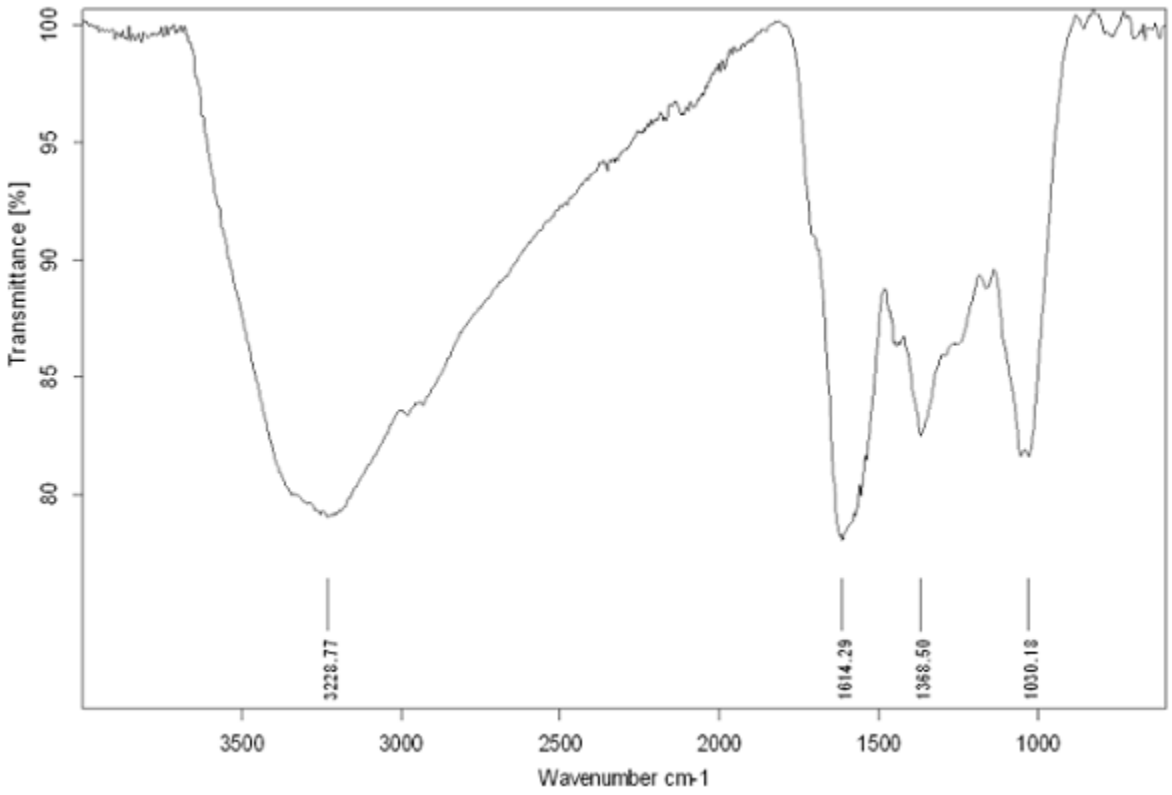

Figure 3.7. FTIR of Amine-Functionalized CG2 (ACG2-2) stirred in EDA for 72 hrs

The spectra looked similar to the CG2 spectra although a peak at 1614 cm^{-1} was observed and could denote an amine attached to the CG2. However, further characterizations would have to be carried out to prove it. One way to prove if the amine got attached to CG2 was to test it for CO_2 capture which is the main objective of this report using TGA. This is because CO_2 capture is susceptible to amine-functionalized CG2.

3.4 CO_2 Capture

Thermogravimetric analysis (TGA) of both ACG2-1 and ACG2-2 were observed under a helium (He) atmosphere The TGA monitors the change in weight to temperature as a function of time. The initial TGA procedure suggested heating a certain amount of ACG2 to 60^0C at a rate of 20^0C/min under flowing He which took about 20 mins and left isothermal for about 20 mins until the sample reached steady

state (constant weight). After which, CO_2 should be introduced into the system and left to proceed for another 20 mins at which steady weight gain should be achieved.

For ACG2-1, a steady state was reached, however a decrease in the weight of the sample was observed which suggested the fact that amine was not attached to ACG2-1. This could be seen in Figure 3.8.

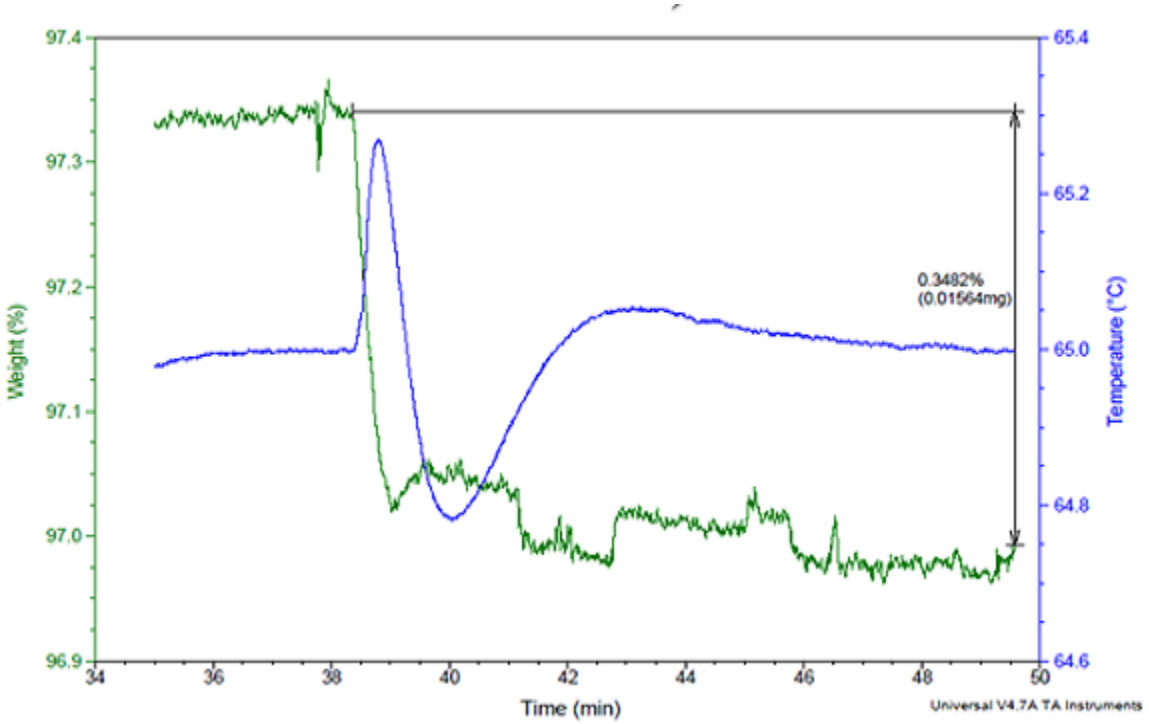

Figure 3.8. TGA of ACG2-1 showing weight loss

As a result of the poor result gotten in ACG2-1, the ACG2-2 was tested for a short amount of time to see if there would be a convincing result of the capture of CO_2 as illustrated in Figure 3.9.

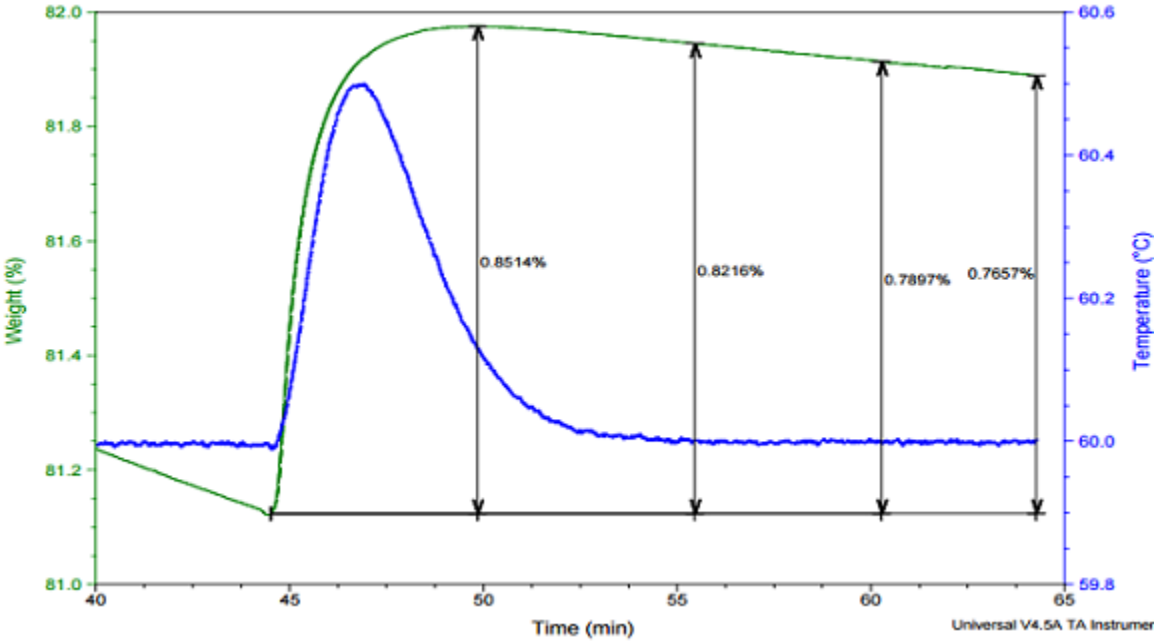

Figure 3.9. TGA of ACG2-2 ran for a short time

A weight gain which corresponded to the removal of CO_2 was observed in that short time which suggested that amine could have been attached to CG2.

To improve the procedure, the sample was left to run for a longer amount of time as seen in Figure 3.10.

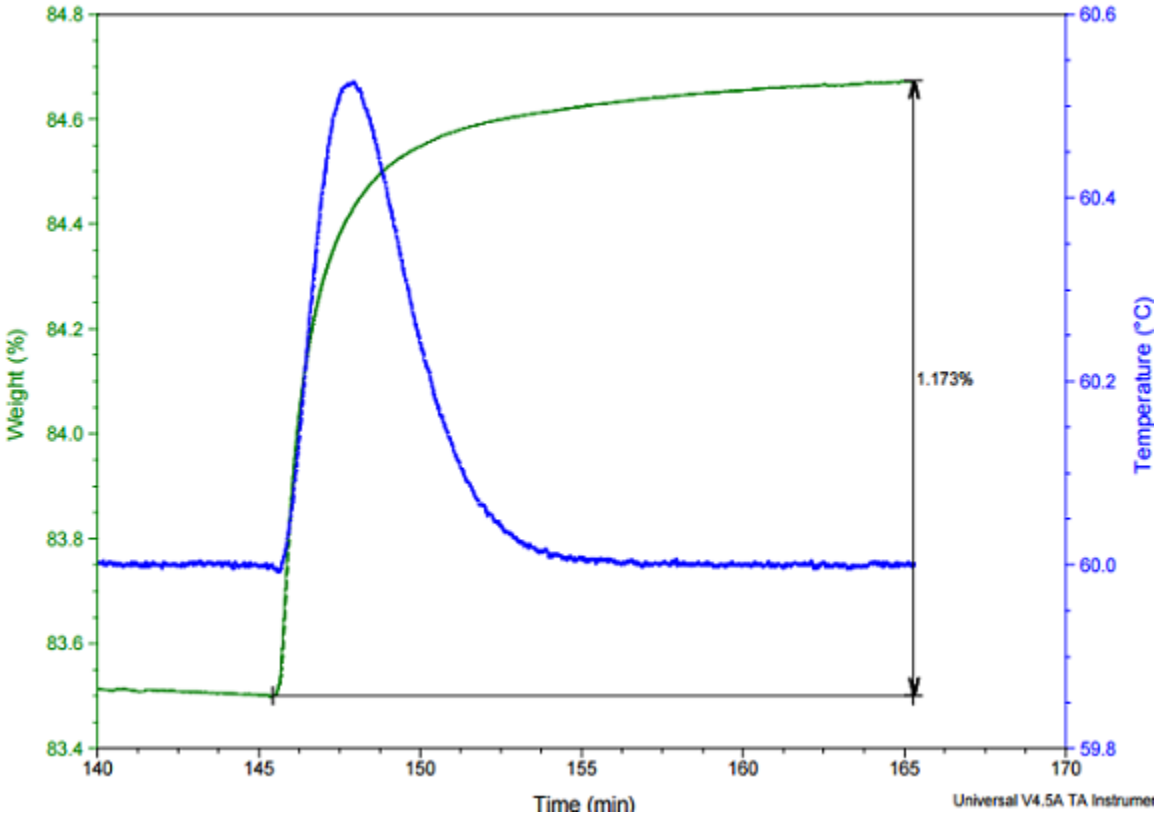

Figure 3.10. TGA of ACG2-2 ran for a longer time

The sample was left to run until it reached a constant weight (at about 145 mins) before switching to CO_2 and left isothermal for 20 mins. It was observed that the weight kept on increasing with time. However, due to the isothermal stage, the sample was not able to reach a steady-state weight gain before it got terminated. As a result, about 1.173% of CO_2 was captured. It follows that if the sample was left to run for a longer time, it may have captured more CO_2 at steady state.

CHAPTER 4

CONCLUSION AND FUTURE WORK

The various functional groups in graphene oxide were investigated and functionalized to aid CO_2 capture. The modified method for producing GO has significant advantages over Hummer's method. It yields a more oxidized GO and proved the existence of the hydroxyl, epoxyl and carboxyl functional groups in its FTIR spectra. The hydroxyl group was subjected to Johnson-Claisen rearrangement conditions to give ester groups which was easily saponified to give carboxyls. Result from FTIR confirms this reaction. An economical approach was used to functionalize CG2 with amines and used for CO_2 adsorption. It can be concluded that amine functionalized CG2 (ACG2-2) captures CO_2 but not a considerable amount as would have been expected (above 13% as suggested by Chen et al.) using this procedures. This could have been due to errors in experimental procedures especially in the amine functionalization, personal or characterization errors. For this reason, improvements in the procedure should be incorporated into future work.

For future work, the method of functionalizing amines via acid chlorides should be investigated. This involves reacting CG2 and oxalyl chloride with the addition of the amine under nitrogen. Extreme caution should be taken during this procedure since it contains toxic substances. Also, different characterization methods like elemental analysis, and BET should be explored in differentiating the functional groups and the surface areas of the samples. Also, a gas chromatography mass spectra could be used instead of the TGA to better test for CO_2 capture. This is because it was found out after this report was finished that the TGA used was not designed to introduce CO_2 into the system, so most of the TGA results are inaccurate. Lastly, every

procedure used in this work could in itself be modified depending on the individual. For instance, the amine could be left to stir for longer with the CG2 and TGA could be designed to introduce CO_2 into the system and could run for longer at different specifications to produce better outcomes.

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